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INFORMATION ON NORESEAL--A NEW CORK SUBSTITUTE

By S. I. Aronovsky, W. F. Talburt, and E. C. Lathrop
Agricultural Residues Division

Northern Regional Research Laboratory
Peoria 5, Illinois

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By S. I. Aronovsky[1], W. F. Talburt[2], and E. C. Lathrop[3]
Northern Regional Research Laboratory[4]
Peoria, Illinois

Natural cork, practically all imported from Spain, Portugal, France, and North Africa, was placed under strict Governmental control in June 1941, at which time about two years' supply of this commodity was on hand in this country. After our entry into the war the increased production tempo of military and Lend-Lease materials greatly aggravated the cork supply situation, and resulted in strong curtailment of its use for the manufacture of civilian goods.

The bottling industries, including producers of soft drinks, beer, food juices and extracts, were quite concerned over the approaching shortage of composition cork for liners in crown caps. The cork for this use normally amounts to more than 30,000 tons annually. Representatives of these industries appealed to the Department of Agriculture for assistance, and research on this project was begun by the Agricultural Residues Division of the Northern Regional Research Laboratory early in 1942.

Crown Liner Specifications

Liners for crown seals must be compressible to withstand the capping pressure and they must have a fair degree of elasticity to allow for deformation without rupture during the capping process. At the same time they must be impermeable to air and other gases in order to withstand the pressure of carbonated beverages, or to hold the vacuum created in packing fruit and vegetable juices and other fluid food products. Finally, the liners must have a fair degree of resilience to resist fatigue for relatively long periods of storage under pressure or vacuum and must not be affected by the temperatures of pasteurization or sterilization used in processing the bottled products. The cork liner for crown caps has a diameter of 1.045 to 1.052 inches and varies in thickness from about 0.10 to 0.12 inch.

1/ Head, Pulp and Paper Section, Agricultural Residues Division.

2/ Present address, Western Regional Research Laboratory, Albany, California.

3/ Chief, Agricultural Residues Division.

4/ This is one of four regional research laboratories operated by the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

For beer, foods, and certain soft drinks it is the practice to cover the surface of the cork liner in contact with the liquid contents of the bottle with a "spot" impervious to the liquid, such as aluminum foil, resin-coated paper, et cetera.

Requirements for a Cork Substitute

The success of natural solid cork and composition cork in meeting these specifications for a liner for crown caps is due mainly to its physical structure. Cork consists of an essentially uniform aggregation of minute hollow plant cells, the contents of the living cells having disappeared while the walls have become thickened and toughened due to the formation of various substances of complex nature. Each of these minute, relatively thick-walled cells is sealed against all other cells so that the entrapped air cannot easily move about or escape. Thus, the compressibility, impermeability to gases, resilience, and resistance to fatigue of natural cork are due to its inherent physical structure.

It logically follows that a successful substitute for cork must have a physical structure similar to that of cork. It is also obvious that when cork is available in sufficient supply, a substitute must not only have properties equal or superior to those of natural cork, but it must also be able to compete with cork on a price basis. Numerous patents have been issued, both here and abroad, for various types of cork substitutes. These cover the use of materials such as cellulose and cellulosic compounds (1), sawdust (2), corncobs (3), hydrated silica (4), and plasticized zein (5). These materials have failed to substitute for cork, even in a period of shortage.

Pith from plant materials has been suggested as a cork substitute. Pith, however, consists of an aggregation of hollow cells having relatively thin, inelastic walls, and as such will not serve the purpose any better than cellulosic fibrous materials. By grinding pith, particles are produced containing but few air cells. If these particles could be successfully surrounded by relatively thick elastic walls, the product should be similar to cork in structure. This is the basic conception in the development of Noreseal.

Noreseal meets all of the required specifications for a liner for crown seals, and, in fact, excels natural cork in some of its properties. Tests on more than 7,000 commercially packed bottles of various beverages and foods have demonstrated the practicability of this material. The technical committee of a large beverage association has named this composition as its first choice from more than 500 cork substitutes tested to date. The raw materials used in preparing Noreseal are nearly all of domestic agricultural origin and easily obtainable at relatively low cost. One of the national trade associations is financing the building of a pilot plant in Peoria, having a capacity of approximately 150,000 liners per hour, to develop cost and manufacturing data.

Preliminary Experiments

In reducing the original conception to practice, peanut-shell pith, ground to pass an 80-mesh screen, was incorporated into a warm fluid mixture of glue, glycerine, glucose, and formaldehyde in about the proportions used for making the well-known printers' lithographic rolls. By the use of a doctor blade the mix was spread on a waxed table top into a sheet about 0.15 inch thick, and allowed to set and dry. While this material, when cut into liner discs, showed considerable resiliency, it was not equal to cork. It was found that a resiliency equal to or better than that of cork could be developed by "whipping" an additional amount of air into the warm mix. Then it was found that by controlling the amount of pith and the amount of "whipped-in" air the degree of resiliency could be regulated.

In order to determine the practicability of the material, several gross of liners cut from the first composition which seemed to have the desired characteristics were submitted to various laboratories of the beverage industry for tests. On the basis of these tests the technical committee of a national beverage association requested that the Laboratory expedite the development, since the product appeared to be of outstanding merit.

Development of Specifications and Physical Tests

By varying the ingredients and methods of preparation a considerable number of compositions were prepared which varied over a wide range of density, resiliency, and other physical properties. Several gross of liners of carefully controlled thickness were cut from each composition, properly spotted with Vinylite-coated paper and submitted to practical tests in a number of breweries. Because the flavor of beer is easily spoiled by contact with soluble or odorous materials and because of the necessity of pasteurization, with the consequent high gas pressure developed, this use of the liner represents a very severe test. Certain of these compositions were prepared by uniformly spreading the warm wet mixture on paper, the latter forming an integral part of the composition and adding greatly to its strength. The advantages of this procedure will be discussed in connection with methods of preparation.

While these practical tests were under way, the same compositions were tested in the laboratory, together with cork liners as controls, for various physical properties such as resiliency, compressibility, density, shear resistance, tensile strength, and elongation. Air-pressure tests were also run on these liners in comparison with cork liners in capping a steel replica of a standard bottle. It was found that those compositions which proved to be the equal of cork in the physical tests would also withstand pressures of approximately 100 pounds in the steel-bottle test.

Two of the physical properties, namely density and compressibility, were found to be correlated with performance. This correlation is shown in figure 1. In bottling tests the compositions within the shaded area, requiring a pressure of 250 to 1,500 lb./sq. in. to obtain 50-percent compression, were all equal to cork liners in performance. These same compositions had a density of 0.57 to 0.68 as compared with water. Compositions outside of this range were unsatisfactory. This finding led directly to a development of manufacturing specifications and larger-scale tests.

The physical tests developed to provide specifications for practical-use data are as follows:

a. Compressibility

The compressibility of the Noreseal composition was determined on a Universal testing machine by pressing a disc between two flat surfaces at a uniform rate of 0.05 foot per second, using a dial gauge calibrated in units of 0.001 inch for measuring the distance between the plates. The required pressures, up to 5,000 p.s.i., to give compressions of 30 to 70 percent of the original thickness were determined. Typical compression data are illustrated in figure 1.

It will be noted from these curves that the compressibility decreased with increasing density of the Noreseal. These data tend to confirm the similarity of Noreseal to cork in physical structure.

b. Shear Value

The test specimen was placed between a steel replica of a standard bottle top and a smooth steel plate. The bottle top, attached to the moving compression crosshead of the testing machine, was lowered with a uniform speed of 0.05 foot per second. The indicated pressure increased uniformly until the specimen failed. The indicated pressure at this point was taken as the shear value of the material. Typical shear values are given in table 1.

The data show that, with decreasing density, the shear value of uncoated Noreseal goes through a minimum at a density of about 0.80. However, even this minimum value is above the pressures encountered in commercial bottle-capping machines. No shearing of the Noreseal liners occurred in any commercial bottling tests.

c. Tensile Strength and Elongation

Tensile strength and elongation were determined with a Schopper paper tensile-strength tester using test specimens 1 inch long and 15 millimeters wide.

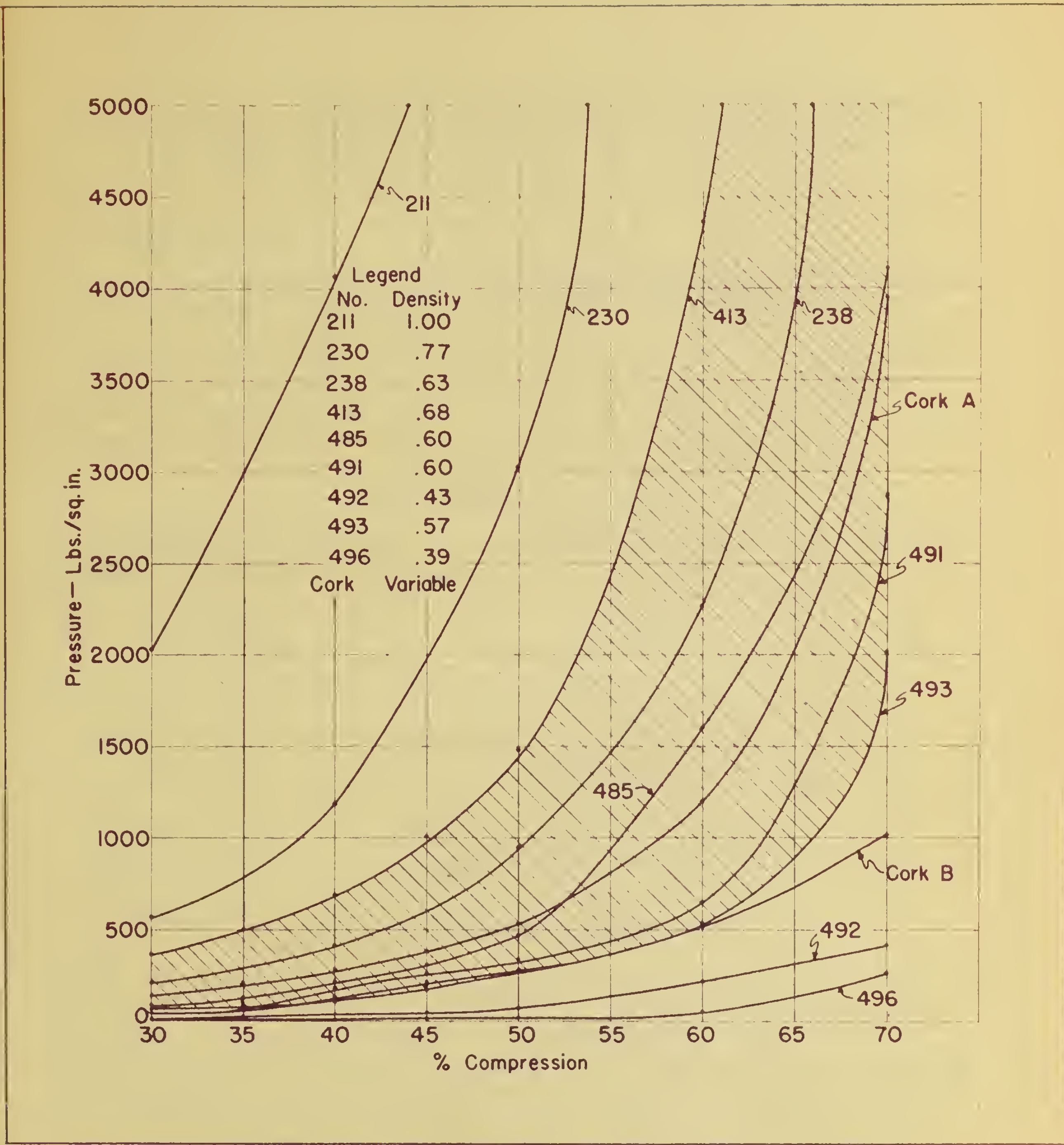

Legend
No. Density
211 1.00
230 .77
238 .63
413 .68
485 .60
491 .60
492 .43
493 .57
496 .39
Cork Variable
Pressure — Lbs./sq. in.
% Compression
Cork A
Cork B
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
30
35
40
45
50
55
60
65
70

Figure 1

The results given in table 2 show that the tensile strength decreases and the percentage elongation increases with decreasing density for Noreseal in sheet form, with or without paper backing.

d. Density

Density of the Noreseal compositions in sheet form was determined by direct weighing of portions of known area and thickness. In the case of Noreseal poured directly into the crowns, its density was determined indirectly from the loss of weight in xylene.

e. Water Resistance

Noreseal is not dissolved by water. However, prolonged contact with water causes it to swell, with a consequent loss in strength, and also results in partial removal of water-soluble constituents. Where Noreseal is to be used for sealing aqueous liquids, it is therefore necessary to cover the side next to the liquid with a suitable water-impervious material, such as the "spot" now frequently used on cork. Noreseal is not affected appreciably by oils, hydrocarbons, or fats.

Preparation of Noreseal

A representative formula for the preparation of Noreseal has the following ingredients and proportions:

Function	Substance	Parts
Elastomer material	Glue or gelatin	100
Plasticizer	Glycerine	75
	Glucose	75
Filler	Ground peanut hulls	100
Liquid medium	Water	350
Foaming agent	Saponin or sulfonated hydrocarbon	1-2
Setting agent	Formaldehyde or a material that liberates formaldehyde	2

With the exception of the setting agent, the order of adding these ingredients is not critical. The glue may be soaked in water and then dispersed by heating before the other materials are added, or the water and plasticizer may be added in part to the glue and in part to the filler which are then mixed together and heated, or all of the ingredients may be mixed together and then heated. After the mixture is heated to about 60°C. it is stirred at high speed or "whipped" in a mixer, as illustrated in figure 2, until a sufficient amount of air has been incorporated keeping the temperature fairly constant. The setting agent is then stirred into the mixture and the viscous fluid is ready to be formed into sheets or poured into individual crown caps (figure 3).

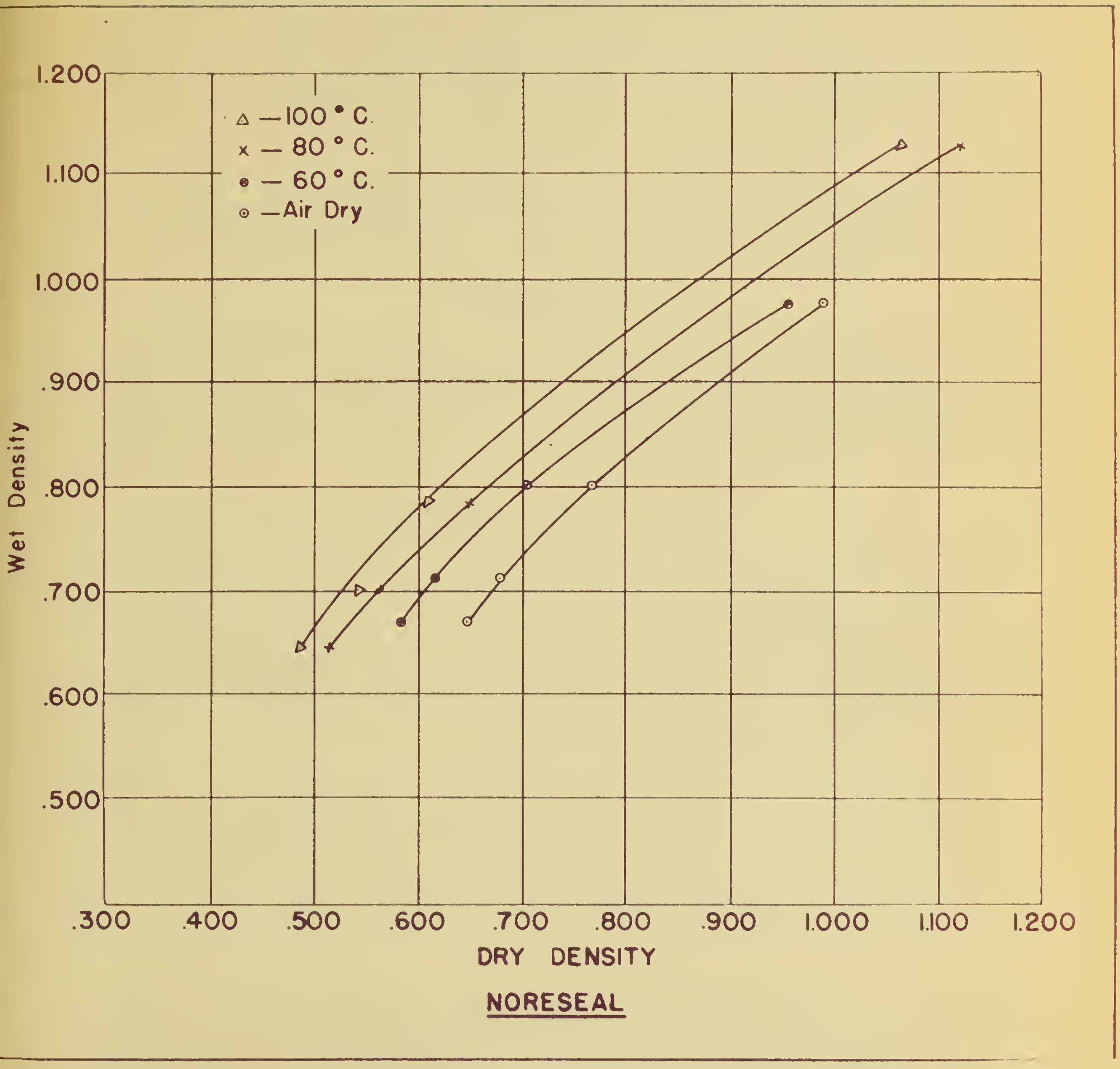
The density of the fluid mixture, just prior to adding the setting agent and pouring, has been found to be the principal controlling factor for governing the properties of the final product. Small variations in the proportions of the various ingredients have but little effect, since they may be compensated for by the amount of air whipped into the mixture. The relationship of the density of the fluid material to that of the final product, dried at different temperatures, is shown in figure 4. After drying, the samples were conditioned at 70°F. and 50 percent relative humidity before their densities were determined. It is evident from these curves that, within the practical working range of the Noreseal for crown capliners, the wet-dry density relationship is almost linear. For a given wet density the dry density decreased with increased temperature of drying, probably due to the greater expansion of the air cells within the compositions at the higher temperatures. The drying, except for those samples dried under normal room conditions, was carried out in ovens with circulating air. The wet density determination is an important manufacturing control test.

The viscous fluid was poured into a rectangular frame at one end of which was a doctor blade set at the desired height; as this instrument was drawn along a flat surface, previously coated with paraffin or other wax, the composition was left behind in the form of a sheet. This method of operation, together with the instrument used, is illustrated in figure 2. The surface of the sheet lost its "tack" in about 5 minutes. After a brief period depending upon the proportion of the various ingredients and upon the amount and type of setting agent used, the sheet was sufficiently strong to be peeled from the surface and placed in a drying oven.

If the composition is coated on a sheet of paper, the latter becomes an integral part of the composition and adds greatly to its strength. The paper may be used wet or dry. Any type of paper capable of being wetted by the composition may be used, including wrapping, newsprint, glassine, parchment, et cetera. Any other type of reinforcing sheet that exhibits a sufficient degree of capillarity with water, such as cotton, wool, silk, or other fabric may be used in place of paper to form a strong bond with the composition. If the composition is coated on a solid material having a flat, waxed or varnished surface, or on paper or fabric coated with Vinylite, ethyl cellulose, or other resin, or on moistureproof cellophane or similar water-repellent material, the composition may be easily peeled, after setting or drying, from the nonwettable material. Furthermore, the side of the composition that was next to the nonwettable surface will show an exact impression of that surface. This indicates the possibility of forming composition sheets with surfaces that match various textures or structural patterns in reverse.

FIGURE 2. Laboratory preparation of Noreseal sheets

FIGURE 3. Noreseal liner materials. Lower left, punched sheet stock. Lower center, punched liners in and out of crowns. Lower right, roll of sheet liner. Upper left, device for centering "spot." Upper center, poured liners with and without "spot." Upper right, rod stock and liners.



1.200
1.100
1.000
.900
.800
.700
.600
.500
Wet Density
△ —100 ° C.
x — 80 ° C.
● — 60 ° C.
⊙ —Air Dry
.300
.400
.500
.600
.700
.800
.900
1.000
1.100
1.200
DRY DENSITY
NORESEAL

Figure 4

Practical bottling tests, to be described later, indicated that the optimum thickness of the finished liner was in the range 0.070 to 0.090 inch in the preferred density range. This required a doctor opening of 0.130 to 0.150 inch, since the shrinkage in thickness on drying amounts to approximately 40 percent. However, there is no general direct relationship between the doctor opening and dry thickness because the shrinkage characteristics are affected by the wet density, by the viscosity, by the speed of coating, by the amount of water to be evaporated, and by the rate of drying. It is evident, therefore, that the doctor-blade setting needs to be correlated with changes in these factors to give sheets having the desired dry thickness.

Crown-cap liners, 1.050 inch in diameter, were die-punched from the sheets, with and without reinforcing backing, for determining the physical properties of these compositions and for bottling tests.

Raw Materials

A large number of compositions, representing various combinations and proportions of different elastomers, setting agents, plasticizers, fillers, and foaming agents, were prepared and evaluated by physical tests and practical bottling tests to determine the most satisfactory materials and procedures.

a. Pith Particles and Other Fillers

Ground peanut shells, bagasse pith, and cornstalk pith were the most satisfactory fillers used in the development of Noreseal. Ground brewers' and distillers' grains were found to be acceptable but, since a ready market for these materials in livestock feeds is already available, they were not considered seriously. Inorganic materials, such as kieselguhr, bentonite and other mineral fillers, resulted in brittle compositions having poor compression characteristics.

Paper pulps from wood had poor flow properties and while cellulose pulps from agricultural residues gave better results, due to their shorter fiber length, even these showed a tendency to flocculate and give uneven flow. Since the principle involved in making Noreseal as a liner for crown seals probably has application in making gaskets and other products, cellulose fibers may prove quite satisfactory in such applications.

Pithy materials are most suitable for the present application and the filler chosen for the bulk of the experiments was ground peanut shell pith because these shells are a readily available waste byproduct of the peanut-oil and confectionery industries. The shells were passed through a hammer mill, and the product was screened through an 80-mesh wire screen. A yield of about 50 percent of satisfactory flour was obtained. As the light pith cells were removed from the peanut hulls,

the residue appeared harder, darker, and more dense. The hard, dark residue has proved to be an excellent abrasive and polishing material, particularly when used with an air blast for cleaning oil and carbon from airplane engines, valves, et cetera. This product has also been found useful as a filler in Noreplast (6).

b. Elastomers

Gelatin, various animal glues, acid and rennet caseins, soybean protein and meal, Vinylite resins, and Norepol (a rubber substitute developed in this Laboratory) were tried as elastomers for compounding Noreseal. Of these, gelatin and the hide glues gave the best results, forming strong and resilient compositions with the lowest tendency toward fatigue. The gelatin and higher-grade glues formed practically odorless products, but the lower-grade glues had odors which were objectionable from the standpoint of use with food or beverage products. The compositions produced with acid or rennet casein and with soybean protein were fairly satisfactory, but not quite as strong or resilient as the gelatin or high-grade hide-glue compositions. However, mixtures of casein or soybean protein with gelatin or hide glue produced compositions similar in their properties to those made with gelatin or hide glue alone. The usual dispersing agents, including urea, ammonium hydroxide, calcium hydroxide, trisodium phosphate, borax, sodium carbonate, and mixtures of such compounds, were used for aqueous dispersion of casein and soybean protein. The Vinylite resins and Norepol were used in the form of aqueous emulsions. The composition sheets made with these materials were not as resilient as those made with gelatin and glue, probably due to the absence, in these experiments, of suitable plasticizers for these resins. The elastomer finally chosen for the major portion of this development was a hide glue with a gel strength of 385 grams. The compositions made with this glue had little or no odor and gave better results than gelatin compositions in practical bottling tests.

c. Plasticizers

The usual plasticizers, humectants, and extenders for glues and gelatin proved to be satisfactory for Noreseal compositions. Glycerine, sorbitol, invert sugar, bland apple sirup, sodium lactate, and ethylene or diethylene glycol were all good plasticizers, either by themselves or in various combinations with each other. Glucose, propylene glycol, and the butylene glycols were not satisfactory plasticizers by themselves, but they could be used to advantage in combination with members of the first group. Sulfonated oils and rosin derivatives increased the water resistance of the Noreseal compositions somewhat, but they had little or no plasticizing or humectant action.

A mixture of glycerine and glucose was selected as the best plasticizer for these compositions in the early experiments. When the supply of glycerine became critical invert sugar was substituted for the glycerine-glucose mixture, with no apparent change in the properties of the final compositions.

d. Foaming Agents

In order to have more air cells in the Noreseal compositions than are contributed by the pithy filler, it is necessary to whip air into the mixture. This is facilitated by the addition of foam-producing and foam-stabilizing agents, such as soap, saponin, alkyl sulfates, sulfonated hydrocarbons, and similar products. Saponin was used in the earlier work with gelatin, but a sulfonated hydrocarbon gave better results when glue, which contains a small amount of oil, was substituted for gelatin.

e. Setting Agents

Various protein-setting agents were tried, including tannin, formaldehyde, paraformaldehyde (trioxymethylene), hexamethylenetetramine, mixtures of formaldehyde with ammonia in different proportions, furfural, metallic chromates, chrome alum, aluminum sulfate, and magnesium lignosulfonate from waste sulfite-pulping liquor. Formaldehyde and its derivatives gave the best results, in the descending order of formaldehyde, paraformaldehyde, and hexamethylenetetramine, with regard to rapidity of action. Paraformaldehyde was chosen as the agent best adapted to the time schedule for laboratory production of the Noreseal compositions.

Final Demonstration of Noreseal Crown-Cap Liners

While other methods, to be discussed later, exist for manufacturing Noreseal crown-cap liners, it was not feasible at the time to consider these methods, because of inability to obtain priorities for building the necessary machinery. It appeared possible, however, to build machinery from low priority materials for producing Noreseal in sheet form in a continuous manufacturing operation.

a. Small Pilot-Plant Machine for Sheet Noreseal Manufacture

1. Forming

In order to demonstrate the possibility of large-scale production of Noreseal in sheet form, a small pilot machine was built from material at hand to form the sheet composition in a continuous or semi-continuous operation. A working sketch of this apparatus is shown in figure 5, and a photograph of the actual set-up in figure 6. The ingredients are heated and mixed in container A (figure 5), then flowed into container B where the mixture is whipped with stirrer R. After

adding the setting agent the viscous fluid is discharged from valve O, through funnel G, into the bay F. Meanwhile, a paper web L from roll C is led under roll D, in container S (for water or other fluid to wet the paper), through squeeze rolls E, over plate P, under bay F, to be coated with the composition M, and then travels under doctor H, onto the belt K, actuated by the roll J. The spray I may be used to spray additional setting agent onto the composition sheet M. The web L with composition M may be detached from the endless belt K and run through driers. If it is desired to make a composition without a reinforcing backing, the belt K may be covered with wax paper or similar water-repellent material, and the composition is then poured into the bay F situated directly over the roll J. The composition may then be peeled, after partial setting, from the water-repellent belt covering, and dried.

The small machine built according to this design worked very well in several trial runs, and illustrated a satisfactory method for the commercial production of the sheet Noreseal.

2. Drying

The sheeted wet material, containing about 55 percent moisture can be dried to 70 percent solids in 2 hours by exposure to circulating air at room temperatures (70°-80°F.). Starting with the wet material, the times required for drying to 85 percent solids in a circulating-air oven at various temperatures are as follows:

Oven temperature °C.	Oven temperature °F.	Time Minutes
60	140	90
70	158	60
80	176	45
90	194	40
100	212	30
110	230	25

This information has been applied in the design of a larger-scale pilot plant.

3. Humidity Equilibrium

Noreseal sheets in equilibrium with air of 50 percent relative humidity at 70°F. contain about 15 percent moisture. It is necessary to keep Noreseal liners in air having a relative humidity between 40 and 70 percent. When exposed to air having a relative humidity lower than 40 percent, Noreseal becomes less resilient and somewhat brittle. However, when Noreseal in equilibrium with air of very low relative humidities is brought into air of a proper degree of humidity, it

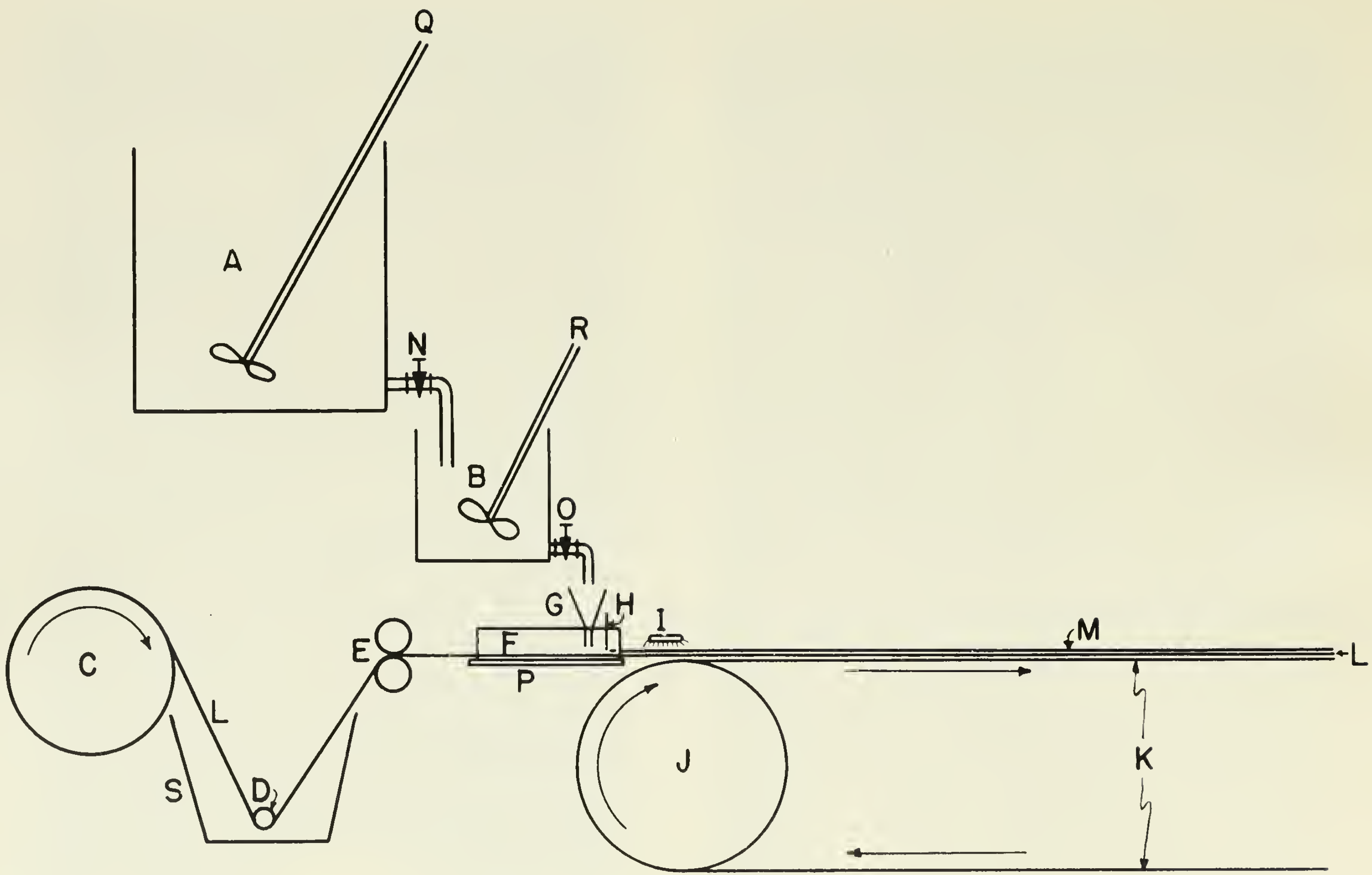


FIGURE 5. Sketch for small pilot machine for demonstrating continuous production of sheet Norseal

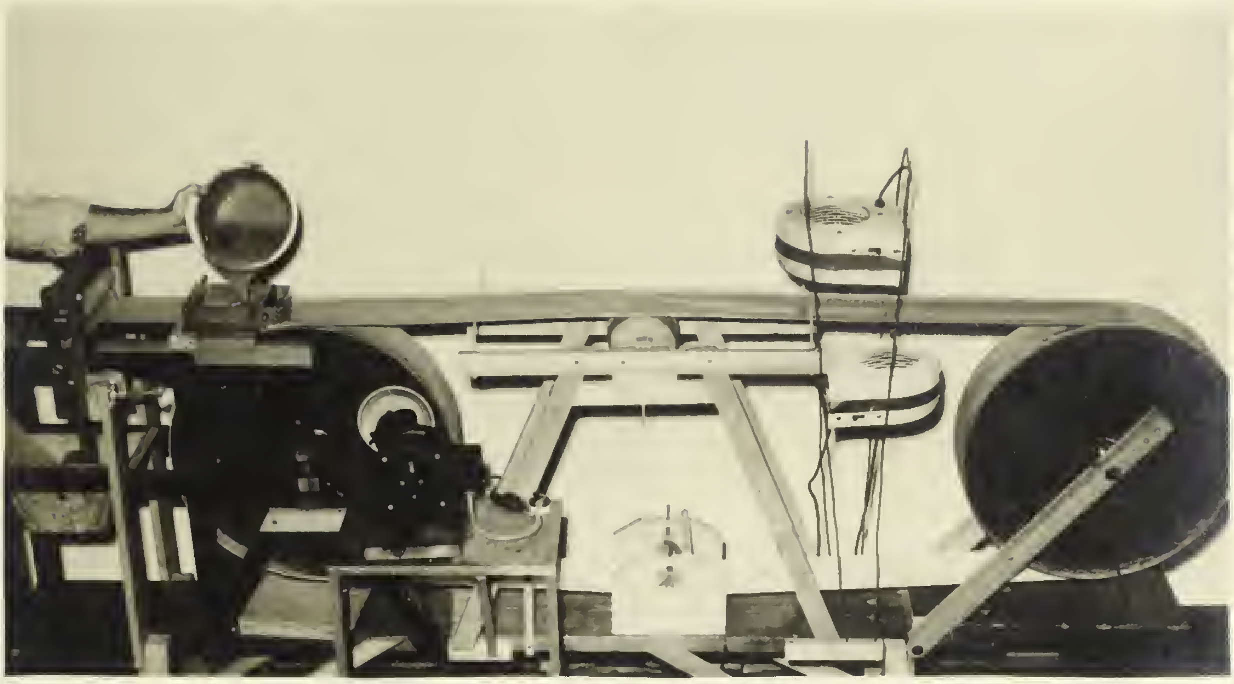


FIGURE 6. Small pilot machine for demonstrating continuous production of sheet Noreseal.

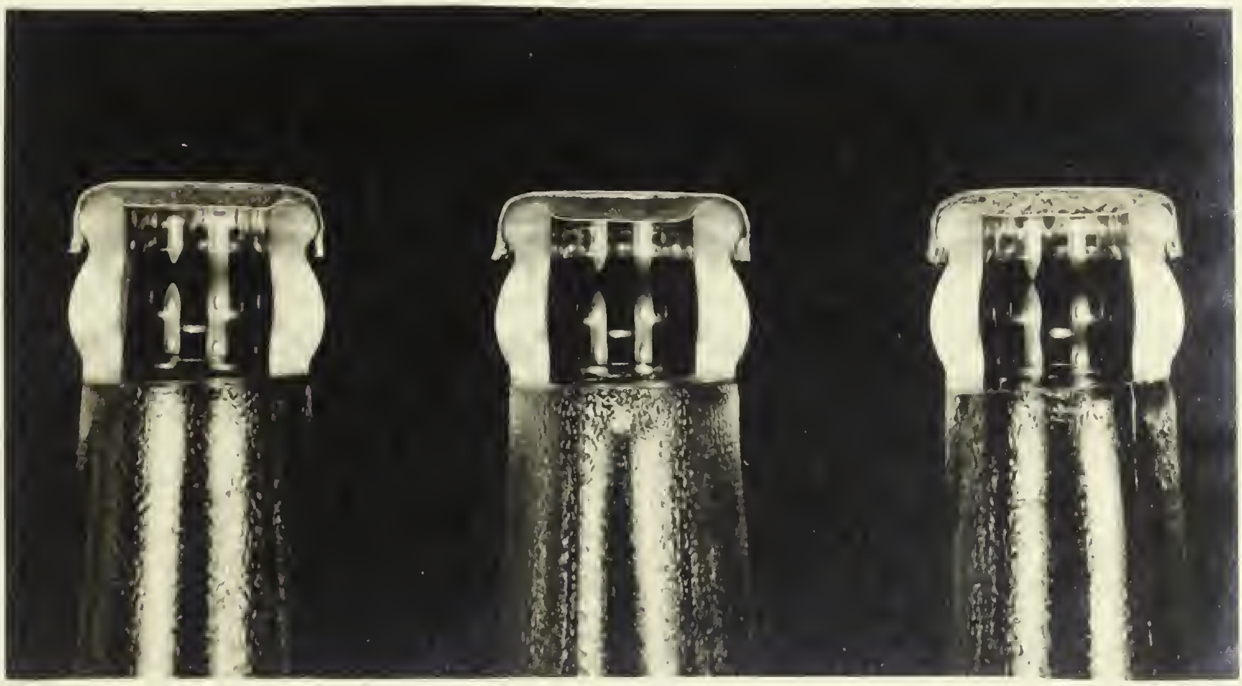


FIGURE 7. Cross-sections of crown-sealed bottle tops. Left, Noreseal sheet liner. Center, poured Noreseal liner. Right, composition cork liner. Note flow of Noreseal liner over bottle lip.

quickly recovers its resiliency. This problem has been discussed with a large number of bottling-plant operators and they say that it does not present any serious difficulty, since bottling plants always maintain relatively high atmospheric humidities. Cork manufacturers likewise agree that the problem can be handled in a satisfactory manner. Indeed, this situation exists to a noticeable, but less critical, degree with composition cork liners.

4. Punching and Spotting Liners

Sheet Noreseal can be cut into liners with regular die-punching machines and the liners can be spotted with the same machines used commercially for spotting cork liners. For making several thousands of liners for the large-scale bottling tests, it was necessary to develop special laboratory tools for accurate punching and spotting.

5. Recovering and Re-Use of Stamping Waste

About 30 percent of stamping waste results from the use of Noreseal in sheet form. A satisfactory method of re-using this scrap consists of grinding the material with water and incorporating it with the regular composition, keeping the proportions of the various ingredients the same as in the original composition. When Noreseal was prepared in this way, using up to 35 percent of stamping waste, little difference could be discerned between these compositions and those made entirely with fresh ingredients. When the scrap is obtained from paper-backed sheet Noreseal, screening is generally required to remove the fibrous material, which tends to flocculate.

b. Other Methods of Producing Noreseal Liners

1. Forming Directly in Crown Caps

The present commercial method of manufacturing crown seals with composition cork consists of producing the disc-shaped liners and then fastening them in the crown caps by means of an adhesive. The same technique must be employed with discs made from Noreseal sheets or rods. With the development of Noreseal, however, an entirely new method of forming crown seals became possible. The viscous, fluid composition from the mixer can be dispersed directly into the crown caps and allowed to dry, thus eliminating the steps of sheet or rod formation, punching or slicing, and also insertion and cementing of the liners into the caps. No waste is produced by this method. Shrinkage of the composition during drying produces a slightly concave surface. After drying, the completed seal is seasoned in air having a relative humidity of 40 to 60 percent before use. The spot material may be placed upon the wet composition and thus avoid the use of an adhesive, or it may be cemented to the dry composition as in the case of the sheet stock. Crown seals produced in this manner

are shown in figure 3. This method holds a great deal of industrial promise, but cannot be used extensively until procurement of the necessary dispensing machinery is made available. Such a method, furthermore, could be carried on at a bottling plant.

2. Forming Noreseal into Rods

The commercial method for producing composition cork-liners consists in grinding the cork, coating it with a mixture of gelatin or glue and plasticizer (equal to about 30 percent of the weight of cork) in a rotating drum, and molding the coated particles into a cylindrical rod from which crown-cap liners of the desired thickness are sliced. Since present cork-liner manufacturers are equipped with machinery for this type of product, it was considered most desirable to try to adapt Noreseal to the present manufacturing process.

Preliminary attempts were made to produce Noreseal particles of the proper granular size directly from the viscous fluid composition. This was accomplished on a laboratory scale by extrusion of the fluid composition into thin strands which were then cut into particles of the desired length. These particles were easily molded into rods of the desired diameter. Final details of this process have not been developed.

Attempts were also made to mold the fluid Noreseal compositions directly into rods but, due to non-uniform shrinkage during drying, they were not successful.

Rods can be formed by shredding the stamping waste from the production of liners from sheet Noreseal to form particles approximately 1/8 inch in size, coating these particles with glue, and molding them into a cylinder of the desired diameter, such as shown in figure 3. Some of the Noreseal scrap was sent to a crown-cap plant for the production of rods in commercial equipment; the report stated that this material behaved as well as cork. This method thus presents a means for the complete re-use of Noreseal waste, without change in the type or operation of commercial equipment available in most crown-cap plants.

Large-Scale Commercial Bottling Tests With Soft Drinks, Beer, and Foods

Using the formulation that had shown the greatest promise in preliminary tests, a number of compositions were prepared which varied with respect to density, resiliency and thickness. These compositions, when used in the practical bottling tests, served to establish manufacturing specifications. In the practical tests it was also determined that the standard-size spot, as compared to a larger one, was preferable. In addition to liners prepared from sheet Noreseal, a sufficiently large lot of poured and spotted crown seals for experimental use was prepared as described above.

More than 7,000 bottles of liquid beverages and foods have been sealed with Noreseal in the operating bottling lines of carbonated beverage and food plants and of breweries. The Noreseal-lined crowns were dumped into the bottling-machine hoppers without interrupting the normal continuous operation of the machines. The sealed bottles were removed after they had received the normal treatments in the various plants, such as pasteurization, rotation, labeling, dropping into cartons, et cetera. As controls, bottles sealed with the usual composition cork-liners, just prior to and immediately after using the Noreseal liners, were taken. The bottled liquids were subjected to various treatments designed to simulate normal and extreme conditions of storage and handling in the average life of these liquids, after which the sealed bottles were tested for pressure, in the case of carbonated beverages, or for vacuum, in the case of hot-bottled foods. Taste tests were made by qualified tasters in the various plants to determine if the taste had been affected by the sealing composition.

Typical results obtained with Noreseal liners punched from sheets are given in table 3. It may be seen from these data that Noreseal is fully equal to cork under the conditions used in these tests. Particular attention is called to the extended hot storage and normal storage tests since they are considered to be quite drastic. The conditions used in these tests are considerably more drastic than those usually encountered in the normal life of the beverage.

The taste of the beer from bottles sealed with Noreseal and subjected to the above tests was fully as good as that obtained with cork-sealed beer. In fact, some of the hot-storaged beers, as well as some stored for the longer periods under normal room conditions, had a greater degree of stability, when sealed with Noreseal. This was particularly true when unspotted composition cork was used in the control tests.

The effects of the density and thickness of sheet Noreseal on the gas pressure in bottled beer are shown in table 4. It is seen from these data that Noreseal with a density of about 0.6 and a thickness of 0.080 to 0.090 inch gave optimum results, under the drastic conditions of hot storage. However, densities as low as 0.4 and as high as 0.8, with correspondingly greater and lesser thickness, respectively, are fully satisfactory for gas retention in beer and in other carbonated beverages. When stored at normal temperatures all of these compositions, with the exception of Nos. 495-2 and 496, gave satisfactory results.

Similar results were obtained with Noreseal liners made by pouring the fluid compositions directly into the crowns, as shown in table 5. No "leakers" were found in this test, either after storage at high temperatures or long storage at normal temperatures. It should be noted that the beer used in this test had a lower initial gas pressure.

The greater efficiency of the poured liner in crown sealing may be due to its concave surface giving a somewhat larger area in contact with the bottle top. This is illustrated in figure 7, where it may be noted that the poured Noreseal liner fills the space between the crown and bottle top more completely than either sheet Noreseal or composition cork.

Tests on carbonated nonalcoholic beverages are shown in table 6. It is seen clearly from these data that Noreseal is fully equal to cork, even at pressures up to five volumes. The low values noted are still higher than necessary for good palatability, and were single tests, as seen from the average values. The flavors of these beverages were normal, as attested by qualified tasters from each of the plants involved.

In bottling noncarbonated, perishable liquid foods, the product is sealed while still hot. On cooling, contraction of the liquid takes place resulting in the creation of a vacuum within the bottle. This vacuum must be maintained for a reasonable period to avoid undue spoilage of the bottle contents. Grape juice was sealed with sheet Noreseal and with composition cork on a commercial machine of a large food processing plant. Some of the Noreseal liners were faced with a full spot, i.e., the liners were completely covered with Vinylite-coated paper (figure 3). After cooling and storage at room temperature for 12 days, tests with a vacuum gauge showed that the bottles had a vacuum of 14 to 17 inches. After 6 months at room temperature, the following results were obtained: In bottles sealed with Noreseal having a standard spot, the vacuum was 14", 14", 15", 15"; with full-spotted Noreseal it was 15", 11", 15", 14"; and with composition cork it was 12", 12". No off-flavors could be detected in any of the bottled grape juice tested. These results indicate that Noreseal is satisfactory for use in the hot bottling and storage of liquid foods.

Noreseal is apparently more resistant than composition cork to attack by mold organisms. After heavy dusting with spores of Aspergillus niger in an atmosphere saturated with water vapor at room temperature, growth of the organism was noted on composition cork at the end of 7 days. Under the same conditions Noreseal resisted the attack for 15 days.

It is thus apparent, from a utilitarian standpoint, that Noreseal is fully equal to composition cork for sealing bottled carbonated beverages, beer, and foods. It has the advantage over the natural product in that its composition and physical properties, such as density, resilience, hardness, color, and shape, may be varied and controlled within relatively wide limits. Furthermore, it can be used in present bottling machinery without any modification of the latter, whatsoever.

Cost Data

The cost of production is always a governing factor in the commercial development of any new material. This includes the cost of the raw material, power, steam, labor, interest on capital investment, depreciation, and other fixed charges, sales promotional work, et cetera. It is not possible to estimate these costs for Noreseal with any degree of accuracy, however, until sufficient operating data on a fair-sized pilot-plant scale have been obtained. Such a pilot plant financed by a national beverage trade association is under construction in Peoria at the present time. However, a comparison of the cost of the raw materials, based on present prices and on known pre-war prices, is given in table 7. It may be seen from these values that the raw material cost is considerably higher now than it was in the normal period. The value of 1.62 cents per gross of liners is based on re-use of the punching waste and a yield of 170 liners per square foot of sheet Noreseal. According to available information, the present price of composition cork is 4.17 cents per gross of liners.

Summary and Conclusions

1. The importance of the physical structure of a substitute for cork for crown sealing of bottled goods is discussed briefly.

2. Noreseal, a new material with physical structure and properties similar to those of natural cork, and produced from relatively low-cost, domestic, and mainly agricultural raw materials, is described.

3. Methods are given for producing Noreseal in the form of sheets and rods, from which crown liners may be stamped or sliced. A small pilot machine for demonstrating continuous or semicontinuous production of sheet Noreseal is described.

4. A novel method for producing the liner directly in the crown was developed, thus eliminating the usual operations of stamping or slicing, and of inserting and cementing the liner into the crown.

5. Specifications for Noreseal, including physical properties such as density and compressibility, were developed for correlation with practical-use data. These physical properties may be varied and controlled over a relatively wide range.

6. Noreseal is more resistant than composition cork to attack by mold organisms.

7. More than 7,000 bottles of carbonated beverages, beer, and foods were sealed with Noreseal on the regular bottling lines of commercial plants and the products were tested for gas pressure or vacuum and for taste.

8. The results of these commercial tests lead to the conclusion that Noreseal is fully equal to composition cork for sealing carbonated beverages, beer, and foods, and that it can be used directly in present commercial bottling machinery.

9. In the near future the process will be studied in a pilot plant having an hourly production rate of 150,000 liners. Cost and manufacturing data, as well as data from full-production bottling tests will be secured.

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The mention of these firms should not be construed as an endorsement of them or their products by the U. S. Department of Agriculture.

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Table 1.- Shear values of Noreseal in comparison with composition cork

Material (thickness, 0.085" ± 0.005")[1]	Density	Shear value (pressure at failure)
Noreseal		Pounds
Without paper backing	1.03	1,100
	0.87	800
	0.65	950
	0.57	1,350
With paper backing	1.08	2,550
	0.65	1,800
	0.50	2,600
Composition cork	0.30	7,500

[1] Variations in thickness from 0.08 to 0.09 had no noticeable effect on the shear value.

Table 2.- Tensile strength and elongation of Noreseal

Material	Density	Thickness	Tensile strength / thickness	Elongation
		Inches	Kg.	Percent
Without paper backing	0.73	0.080	120	30
	0.63	0.070	79	37
With paper backing	0.95	0.064	250	14
	0.52	0.085	100	18

Table 3.- Pressure tests on beer sealed with Noreseal and subjected to various treatments[1]

(Liner density 0.60-0.68, thickness 0.080-0.090")

Liner material	Treatment given sealed bottles	Number of tests	Pressure in volumes of CO_2 per volume of liquid		
			High	Low	Average
Noreseal	None (one day	31	2.80	2.65	2.69
Cork	after bottling)	4	2.71	2.67	2.69
Noreseal	Icing[2]	10	2.77	2.69	2.73
Cork		12	2.85	2.70	2.78
Noreseal	Immersion[3]	12	2.84	2.65	2.75
Cork		12	2.79	2.66	2.74
Noreseal	Dropping[4]	24	2.80	2.56	2.71
Cork		18	2.81	2.65	2.74
Noreseal	Rotation[5]	5	2.77	2.69	2.72
Cork		6	2.79	2.66	2.72
Noreseal	Dry storage[6]	16	2.78	2.65	2.72
Cork		8	2.83	2.58	2.73
Noreseal	Hot storage[7]	26	2.89	2.56	2.75
Cork		17	2.93	2.56	2.72
Noreseal	Normal storage[8]	22	2.75	2.62	2.70
Cork		10	2.78	2.65	2.71

1/ Before applying any other treatment, bottles were carried in a delivery truck for one week.

2/ After trucking, bottles placed in ice for 5 days; ice allowed to melt over week end; ice treatment repeated for a second week.

3/ After trucking, bottles laid on their side and completely immersed in water at room temperature for two weeks.

4/ After trucking, full case of bottles dropped on case containing test bottles from height of 6 inches, every day for 12 days.

5/ After trucking, bottles rotated end over end on wheel turning 0.5 r.p.m. for two weeks at 100°F.

6/ After trucking, bottles stored at 70°-80°F. and approximately 5 percent relative humidity, for one to three weeks.

7/ After trucking, bottles stored in room maintained at 100°-110°F. for 10 to 21 days.

8/ After trucking, bottles stored under normal room conditions for 30 to 90 days.

Table 4.- Effect of density and thickness of Noreseal liners on pressure of beer stored at 100° to 110°F. for 10 to 21 days

Code number of composition	Number of tests	Liner specifications		Pressure in volumes of CO_2 per volume of liquid		
		Density g./cc.	Thickness inches	High	Low	Average
491-1	9	0.604	0.080-0.085	3.00	2.47	2.70
491-2	11	0.604	0.065-0.070	2.92	1.93	2.49
485	26	0.600	0.085-0.090	2.89	2.56	2.75
493	16	0.571	0.065-0.070	2.87	2.21	2.64
495-1	10	0.500	0.070-0.075	2.45	1.50	2.05
495-2	11	0.500	0.060-0.065	2.08	1.50	1.77
496	10	0.386	0.060-0.065	2.48	1.82	2.13
1/	27	2/	2/	2.99	2.65	2.81
Cork	17	---	---	2.93	2.56	2.72
Cork	3/ 4	---	---	2.74	2.70	2.71

1/ A group of all of the liners tested one day after bottling.

2/ Included liners of all of the above densities and thicknesses.

3/ Tested one day after bottling.

Table 5.- Pressure tests on beer sealed with poured Noreseal liners (Density 0.52-0.90, thickness 0.065-0.090")

Liner material	Treatment given sealed bottles	Number of tests	Pressure in volumes of CO_2 per volume of liquid		
			High	Low	Average
Noreseal	None (one day after	62	2.59	2.45	2.52
Cork	bottling)	8	2.62	2.43	2.54
Noreseal	Hot storage 1/	141	2.60	2.35	2.49
Cork		13	2.65	2.43	2.54
Noreseal	Normal storage 2/	151	2.60	2.39	2.48
Cork		18	2.72	2.43	2.53

1/ Stored at 100° to 110°F. for 10 to 21 days after bottling.

2/ Stored under normal room conditions for 30 to 90 days after bottling.

Table 6.- Pressure tests on nonalcoholic carbonated beverages sealed with Noreseal

(Sheet liner density 0.65-0.68, thickness 0.080-0.090")

Beverage bottled and kind of liner used	Pressure in volumes of CO_2 per volume of liquid: Hot-cold storage[1/]				Normal storage[2/]			
	Number of tests	High	Low	Average	Number of tests	High	Low	Average
Cola A, Noreseal	4	3.81	3.68	3.75	10	3.92	3.30	3.71
Cola A, cork	5	3.95	3.80	3.86	6	3.99	3.80	3.89
Cola B, Noreseal	14	3.87	[3/]2.72	3.55	18	3.83	[3/]2.85	3.59
Cola B, cork	6	3.89	3.66	3.75	17	3.80	3.40	3.66
Root beer A, Noreseal	6	2.94	2.77	2.86	14	3.40	[3/]2.56	2.93
Root beer A, cork	6	2.97	2.87	2.92	12	3.04	2.65	2.88
Root beer B, Noreseal					18	3.65	[3/]2.55	3.42
Root beer B, cork					18	3.70	3.33	3.48
White soda, Noreseal	19	5.30	4.26	4.94	25	5.24	4.42	4.92
White soda, cork	5	4.83	3.66	4.41	4	5.09	4.62	4.83

1/ Two days at 100°F. and 1 day at 40°F., alternately, for 10 days. A few of the bottles were kept at 100°F. for the entire period.

2/ Ten days at normal room temperature, 70°-80°F.

3/ Only low value in series.

Table 7.- Noreseal raw material costs

Material	Cost per pound 1943	Cost per pound 1939	Noreseal composition	Estimated cost of Noreseal: Per pound 1943	Per pound 1939	Per sq. foot, 0.085" thick 1943	Per sq. foot, 0.085" thick 1939	Per gross of liners 1943	Per gross of liners 1939
	$	$	%	$	$	$	$	$	$
Glue	0.300	0.200	22.7	0.0681	0.0454	0.0180	0.0120	0.0152	0.0102
Peanut hull flour	0.005	0.005	22.7	0.0011	0.0011	0.0003	0.0003	0.0003	0.0003
Invert sugar	0.100	0.060	38.7	0.0387	0.0232	0.0103	0.0061	0.0087	0.0052
Foaming agent	0.150	0.150	0.45	0.0009	0.0009	0.0002	0.0002	0.0002	0.0002
Paraformaldehyde	0.230	0.340	0.45	0.0010	0.0015	0.0003	0.0004	0.0003	0.0003
				0.1098	0.0721	0.0291	0.0190	0.0247	0.0162